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STUDIES OF THERMIONIC MATERIALS  
FOR SPACE POWER APPLICATIONS

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Work done by

Members of the Thermionic  
Direct Conversion Group and  
the Fission Product Study Group

The work accomplished under Contract NAS 3-4165 in January, 1964 is summarized in this report.

## 1. Fabrication Development

### 1.1 UC - ZrC Fuels

#### 1.1.1 Pore Control in UC - ZrC Fuel Specimens

Specimens of UC, 90 UC - 10 ZrC and 30 UC - 70 ZrC are being prepared from the following powder fractions:  $-65\mu/+20\mu$ ,  $-45\mu/+20\mu$ , and  $-45\mu/+10\mu$ . Measurements of the density and BET surface area of the specimens are being made after isostatic pressing and sintering. Results of BET measurements made on such specimens during the month are listed in Table 1.1. Results indicate that use of a  $-45\mu/+20\mu$  powder fraction yields specimens of 83 to 85% of T.D. having a desirable open type pore structure with roughness factors ranging from 188 to 360. This powder fraction is recommended for use in preparation of irradiation specimens.

Some consideration has been given to the ease with which fission gas could diffuse through a large, bulky fuel specimen in a thermionic emitter vented only at the top. If the fuel were to expand and form a tight fit into the emitter cladding, in effect this would seal pores at the cylindrical surface of the fuel. Fission gas would then have to migrate through the fuel length until it could reach the top surface. To increase the ease with which fission gas release could occur, it is proposed that an axial hole be cut in the specimen permitting free access of the fission gas to the venting space after only a short migration through the fuel. To measure the increase in true surface area produced by drilling such a hole the specimen corresponding to the first

entry in Table I had a  $1/4$ " axial hole drilled through it. This increased the geometric surface area by a factor of 1.3 while the true surface area was increased a factor of 1.57 (since roughness factor is the true area over geometric area) as indicated by the second entry in Table I. This indicates that the improvement in the ease with which fission gas can be released went up even greater than expected. In the study of the effect of particle size on the true surface area for the different compositions, a 90 UC-10 ZrC specimen had previously been prepared using a  $-65\mu/+20\mu$  powder fraction. Difficulty has been encountered in pressing specimens of UC and 30 UC-70 ZrC using  $-65\mu/+20\mu$  powder fraction in the existing die using presently available equipment; the pressure applied ( $\sim 97,000$  psi) is not high enough and the compacts often crumble. A large capacity press and a smaller diameter die have been ordered and it is expected better results with the coarse powder fraction will be obtained.

#### 1.1.2 Isostatic Pressing

##### Pressing Medium

Mobay multrathane was tried as an isostatic pressing medium but the material is more difficult to mold than chemosol and less satisfactory compaction is obtained. Use of the chemosol 5045 medium will be continued.

##### Isostatic Pressing of Cermets

Isostatic cold pressing of W-60V/ $oUO_2$  cermets was tried, but was not found to be desirable. Isostatic cold pressing (at  $\sim 97,000$  psi) yielded a specimen which had a dense outer shell but an extremely porous interior. The material near the outside moves much more during pressing

than the material near the center. The outer portion becomes compacted and cold welds into a strong shell which prevents further compaction of the material near the center. UC-ZrC materials on the other hand do not become cold welded but the particles become only mechanically interlocked during compaction. It is possible higher pressures applied during isostatic cold pressing will result in more suitable cermet compacts, but it is believed isostatically hot pressed cermets would be better since plastic flow would occur more readily.

Conventional uniaxial hot pressing has been applied to the preparation of specimens containing tungsten coated UC<sub>2</sub> particles: One specimen was pressed using only vapor deposited tungsten coated UO<sub>2</sub> particles in the loading and a density of 10.4gm/cc was obtained. Another specimen was hot pressed with a charge of the same material as above to which 10w/o tungsten powder was added and blended with the particles. The hot pressed density of this specimen was 11.7g/cc. The above specimen had a length to diameter ratio (L/D) of ~1 or 1 1/2 to 1. Additional specimens will be pressed of the same composition but with an L/D of 1/2 (or less) to 1 in an attempt to increase the density. Metallographic evaluation of the above specimens will be made.

#### 1.2 Vapor-deposited Tungsten

The larger capacity ejector exhaust pump for the vapor deposition apparatus has been received and final assembly of the apparatus is now being completed. Deposition of tungsten under carefully controlled conditions will be started shortly.

Much effort has been expended this month in a joint effort with San Fernando Laboratory to solve problems associated with seal coating

carbide specimens. Although it was thought that control of this problem was attained during the Spring of 1963, attack during the coating process has again been plaguing us. Initial investigation has shown that the bottle of  $WF_6$  being used as feed material was severely contaminated with HF. A purification procedure suggested by personnel at Oak Ridge National Laboratory was attempted. Cooling the  $WF_6$  bottle to  $-76^{\circ}C$  freezes the  $WF_6$  causing it to sink below any HF (in liquid form) which is present. Pumping on the bottle then removes HF preferentially. This procedure was used to purify the  $WF_6$  employed in the seal coat process. During the pumping considerable amounts of effluent were collected and tentatively identified as HF.

The attack during seal coating was lessened by purification of the HF but was not eliminated. Other variables being investigated are:

- (a) Substrate conditions---the specimens now being coated are more porous and closer to stoichiometry than those previously coated.
- (b) Closer temperature control.
- (c) Fluid dynamics of system.

It is expected that the R and D work now being conducted with San Fernando Laboratory will permit a systematic investigation of these variables and lead to a speedy solution of this problem.

Meanwhile, the question of  $WF_6$  purity is of great importance to the program at GA, both from the standpoint of seal coating and also with respect to the influence impurities in the  $WF_6$  will have on the purity and properties of deposited tungsten. Chemical procedures are being established for  $WF_6$  analysis and an analysis of metallic impurities was obtained from one of the bottles of  $WF_6$  at our laboratory (see Table O-2). Efforts are being expended on more thorough investigation of this problem.

## 2. Studies of Properties of High-Temperature Thermionic Materials

### 2.1 Rate of Vaporization of UC-ZrC as a Function of Pore Structures

Measurements of the Langmuir rate of vaporization of sample  $A_2-1$  (29.4 UC-70.6 ZrC, 77.6% dense) have been partially completed. The results obtained in the temperature range 2093-2284°K are shown in Table 2-1. The observed rates for this sample are lower than one would expect on the basis of the results obtained for low density 30 UC-70 ZrC samples under Contract NAS 3-2352.<sup>(1)</sup> It may be recalled<sup>(2)</sup> that the surface roughness factor of  $A_2-1$  is 274, which is about the same as that of the 84.9% dense 30 UC-70 ZrC sample  $A_3$ . It is therefore possible that the open pore fraction of this sample is low even though its density is only 77.6% of the theoretical value. Mercury porosimetric studies will be made on  $A_2-2$  after the completion of the vaporization studies to clarify this point.

The ethane source has been incorporated into the low pressure gas absorption system. Measurements of the surface roughness factor of  $A_1-1$  (30 UC-70 ZrC, 97% dense) will be made shortly.

### 2.2 Fission Product Release from UC-ZrC

Previously, measurements have been made on the following 30 UC-70 ZrC samples:  $A_1-2$  (97% dense, 1800°C),  $A_1-3$  (97% dense, 1900°C),  $A_2-2$  (77.8% dense, 1800°C). During this month, measurements of the fission product release characteristics have been made at 1600°C on sample  $A_2-3$  which was obtained from the same carbide cylinder as  $A_2-2$ . The results are shown in Table 2-2 and Figure 2-1.

The total fractional releases of  $Xe^{133}$ ,  $I^{131}$ ,  $Te^{132}$ , and  $Ba^{140}$  from sample  $A_2-3$  after 67 hours at 1600°C, as compared with that for

sample A<sub>2</sub>-2 at 1800°C in the same period of time (interpolated from Fig. 2-2 of December 1963 report)<sup>(2)</sup> are as follows:

	<u>Xe<sup>133</sup></u>	<u>I<sup>131</sup></u>	<u>Te<sup>132</sup></u>	<u>Ba<sup>140</sup></u>
A <sub>2</sub> -2 (1800°C)	0.44	0.37	0.64	0.08
A <sub>2</sub> -3 (1600°C)	0.16	0.25	0.62	0.027

Thus the releases of Xe<sup>133</sup>, I<sup>131</sup>, Ba<sup>140</sup> from A<sub>2</sub>-3 are less than that from A<sub>2</sub>-2, while the release of Te<sup>132</sup> does not seem to be effected by temperature. The Xe<sup>133</sup> curve in Fig. 2-1 shows an initial high release rate each time the sample was brought back to temperature after it was cooled down for the sampling of the condensate on the cold finger. The reason for this behavior is not understood at this moment. The calculation of the release rate will not be made in the future, since they vary with the time at which the rates are calculated. Instead, attempts will be made to evaluate the diffusion constants from the release data for the correlation of the results obtained from various samples at different temperatures.

Measurements of the fission product release from the 84.9% dense, 30 UC-70 ZrC samples have been started. The first sample A<sub>3</sub>-2 will be studied at 1800°C.

### 2.3 Fission Product Diffusion Through W-Clad

The cell and the measuring instruments are ready to receive sample. The counting efficiency of the scintillation crystal has been calibrated with standard Xe<sup>133</sup>. The experiment is held up because of the difficulties encountered at San Fernando Laboratory of the seal-coating of carbide samples. This problem is currently being looked into by the fabrication group (see Section 1.2). As soon as a sample is available, the cell and the counting equipment will be tested, and the measurements started.



## 2.4 Fuel-Clad Gross Diffusion Studies

The status of fuel-clad gross diffusion studies is summarized in Table 2-3, and the details are described below.

- (a) Run D<sub>2</sub> (30 UC-70 ZrC versus Mo, Ta, Nb, at 1600°C for 50 hours)

Reaction layers were found at the interfaces in all cases.

Liquid phase formation was noted at Mo grain boundaries near the interface. Also, the depth of the reaction zones has penetrated into Ta and Nb for 0.004 inch and 0.01 inch respectively.

- (b) Run D<sub>3</sub> (30 UC-70 ZrC versus Mo, Ir, Re, W-25 Re at 1600°C for 50 hours)

Reaction layer was observed at the interface between Ir and 30 UC-70 ZrC and that between Re and 30 UC-70 ZrC. No particular reaction was observed at the interface between 30 UC-70 ZrC and W-25 Re; however, there was a second phase noted within the W-25 Re alloy. A slight formation of liquid phase was detected at the interface between 30 UC-70 ZrC and Mo, and the grain boundaries of Mo were broadened near the interface. Microprobe studies of the (W-25 Re) - (30 UC-70 ZrC) and Mo - (30 UC-70 ZrC) samples are being carried out.

- (c) Run D<sub>4</sub> (UC versus Mo, Nb, Ta, and Re at 1600°C for 50 hours)

Reaction layers were noted at the interfaces between UC and Ta, Nb, and Re. No reaction layer was observed at the UC-Mo interface, in contrast to the liquid phase formation observed at the (30 UC-70 ZrC) - Mo interface. Grain boundary broadening at the interface, however, was evident. These samples are now undergoing microprobe examination.

- (d) Run D<sub>5</sub> (30 UC-70 ZrC versus Mo and Re at 1600°C for 100 hours)

This run was carried out for two purposes. First, slightly carbon rich 30 UC-70 ZrC was used in order to find out whether the interaction between Mo and 30 UC-70 ZrC observed in Run D<sub>3</sub> was due to the slightly metal rich condition of the 30 UC-70 ZrC used. Second, the test was made in the absence of Ir in order to find out whether the interaction between Re and 30 UC-70 ZrC observed in Run D<sub>3</sub> could be caused by the presence of Ir as a neighbor of the Re during the test, since the Ir reacted strongly with the 30 UC-70 ZrC. The diffusion run has been completed and the samples are waiting to be examined.

- (e) Run D<sub>6</sub> (30 UC-70 ZrC versus Ta, Nb and Mo at 1400°C for 100 hours)

The slightly carbon rich 30 UC-70 ZrC used is the same as that for Run D<sub>5</sub>. This run differs from Run D<sub>2</sub> in that the 30 UC-70 ZrC used is slightly carbon rich rather than slightly metal rich, and that the temperature of the test is 1400°C rather than 1600°C. The diffusion run has been completed and the samples are waiting for metallographical examination.

## 2.5 Fuel-Clad Diffusion-Emission Studies

Studies have been made during this month on sample E<sub>2</sub>, E<sub>3</sub> and E<sub>4</sub>, which are UC (4.90 wt % C) clad with vapor-deposited W (20 mils thick), Re (12 mils thick), and W-25 Re (14 mils thick) respectively. The UC wafers were seal-coated with 2 mils of vapor-deposited W in each case. Studies on E<sub>2</sub> and E<sub>4</sub> are being continued toward the 1000 hour goal at 1800°C; while studies on E<sub>3</sub> have been terminated because excessive changes in emission has been observed after only a very short period of time at 1800°C.

The thermal history plots for  $E_2$ ,  $E_3$  and  $E_4$  are shown in Fig. 2-2, 2-3 and 2-4. Selected Schottky plots for these samples are shown in Fig. 2-5 to 2-10. The results can be summarized as follows.

(a)  $E_2$  (vapor-deposited W clad UC, 4.90 wt % C)

The vacuum emission characteristics of  $E_2$  at  $1800^\circ\text{C}$  change gradually with time (Fig. 2-5). After a few hundred hours at  $1800^\circ\text{C}$ , the Schottky plot consisted of a plateau with emission about a few times higher than that of W at  $1800^\circ\text{C}$ , and another branch beyond the plateau where the emission increased continuously with the increase of field strength. Both the plateau and the branch beyond the plateau shifted toward the abscissa as the residence time of the sample at  $1800^\circ\text{C}$  increased.

When the sample was cooled to  $1400^\circ\text{C}$  (Fig. 2-6), the Schottky plots obtained were all saddle-shaped. The depth of the saddle, however, decreased with the increase of the residence time of the sample at  $1800^\circ\text{C}$ . The emission near the saddle region after the sample was 898 hours at  $1800^\circ\text{C}$ , was still about several times higher than that of W at  $1400^\circ\text{C}$ . It seems that the emission characteristics of the sample both at  $1400^\circ\text{C}$  and at  $1800^\circ\text{C}$  are gradually approaching a certain stable form. The test is being continued.

(b)  $E_3$  (Re clad UC, 4.90 wt % C)

The Schottky plots at  $1800^\circ\text{C}$  (Fig. 2-7) shifted first toward the abscissa and then away from the abscissa as the residence time of the sample at  $1800^\circ\text{C}$  increased. At its lowest position shown in Fig. 2-7, the zero-field emission for the plateau region

of the plot was about ten times higher than that of pure Re at 1800°C. When the sample was cooled to 1400°C, abnormal Schottky plots were obtained (Fig. 2-8). The shape of these plots, however, did not seem to be affected by the residence time of the sample at 1800°C. The test is being continued.

(c)  $E_4$  (W-25 Re clad UC, 4.90 wt % C)

The Schottky plots obtained at 1800°C have abnormal shapes (Fig. 2-9). The emission of the sample increased with the residence time of the sample at 1800°C, and was much higher than that of Re or W. When the sample was cooled to 1400°C, the Schottky plots obtained (Fig. 2-10) were not only abnormal in shape, but also changed with time, indicating a continuous adjustment of surface compositions by diffusion from the bulk. The test was terminated after the sample was at 1800°C for a total of 84-1/2 hours. Sample  $E_4$  is now waiting to be examined in the emission microscope before being subject to metallographical and electron microprobe studies.

Following these samples, the cases to be studied are: vapor-deposited W clad 80 UC-20 ZrC at 1800°C, vapor-deposited W clad 50 UC-50 ZrC at 1800°C and vapor-deposited W clad UC (4.90 wt % C) at 1650°C. Preparation of these samples will be started as soon as the seal-coating difficulties encountered at San Fernando Laboratory are solved. Further studies of Re and W-25 Re clad fuel samples are withheld until the gross diffusion studies of these samples are completed.

## 2.6 Refractory Metal Interdiffusion

The status of refractory metal interdiffusion is summarized in Table 2-4. Runs  $F_1$ , and  $F_2$  consisted of refractory metal couples included in the tantalum diffusion capsule used for fuel-clad gross diffusion studies when space was available. Preliminary examinations have been made on samples of Run  $F_1$ ; the results have been reported in the December 1963 monthly report.<sup>(2)</sup> Samples of Run  $F_2$  are being examined by electron microprobe. Run  $F_3$  is the first all refractory metals interdiffusion run. Although the run was completed, it was found that when the capsule was disassembled, the adjacent metal samples did not make good contact with the tantalum screw located at the top of the diffusion capsule. It became apparent that the total thermal expansion of the column of metal samples was less than that of the tantalum capsule. The contacts between the neighboring pieces were thus lost when the capsule was tested. In the future, UC or UC/ZrC pieces of thermal expansion coefficients higher than that of tantalum will be added to each refractory metal diffusion capsule to compensate for such differential thermal expansion.

The samples of Run  $F_3$  will be kept as controls for establishing the micro-structure of such well annealed refractory metals for comparison with the structures of future refractory metal diffusion samples.

## 2.7 Refractory Metals Diffusion-Emission Studies

To set the reference line for the diffusion-emission studies of Re-W and Ir-W duplex emitters, the vacuum emission characteristics of a vapor-deposited W disc (Sample  $G_1$  0.570 inch diameter and 0.062 inch thick) are being studied at 1800°C in the diffusion-emission cell. The

results obtained to date are shown in Fig. 2-11. It can be seen that after 45-1/2 hours at 1800°C, the shape of the Schottky plot still has not approached its normal form. The test is being continued at 1800°C.

As a comparison, similar studies were made on a cast W sample (sample G<sub>2</sub> 0.570 inch diameter, 1/8 inch thick) at 1800°C. The results are shown in Fig. 2-12. It can be seen that no normal Schottky plot could be obtained even after a few hundred hours at 1800°C, and that the emission was much higher than that of W at 1800°C. It is interesting to note that when the temperature of the sample was raised to 2000°C for 8 hours and then brought back to 1800°C, the emission was much lower than that before such a thermal treatment. (Compare curve B and curve C of Fig. 2-12). However, when the sample was aged at 1800°C for 110 hours, the emission characteristic of the sample returned to that before the thermal treatment at 2000°C. This indicates that some impurities in the cast W which can diffuse to its surface from the bulk, are responsible for its abnormal emission. The impurity contents of both the vapor-deposited W and the cast W are being analyzed.

A diffusion-emission sample consisting of 20 mils of vapor-deposited Re over 60 mils of vapor-deposited W has been received from San Fernando Laboratory. However, when discs of the required size were cut from the disc by the electrical-discharge machining techniques, the Re became detached from the W. Three W discs were recovered, which will be sent back to San Fernando Laboratory to be re-coated with about 10 mils of vapor-deposited Re.

## 2.8 High-Temperature Mechanical Properties of UC-ZrC

The testing furnace was assembled and leak checked. Several

test runs of the specimen loading device were conducted. High temperature mechanical property measurements are scheduled for the coming month.

## 2.9 Emission Microscopy of W-(Uranium-Containing Carbide) Cermets

Previously (3) the emission patterns of a W-UO<sub>2</sub> cermet containing 60 vol % UO<sub>2</sub> were examined at 1650°C in an emission microscope. The results showed that higher work function areas in the W-matrix could co-exist with the lower work function UO<sub>2</sub> dispersions. During this month, a W-UC cermet containing 60 vol % UC was examined at 1300-1550°C. The pattern showed that the UC regions emitted much better than the surrounding W-matrix. However, the W-matrix seems to emit much better than that of pure W. It appears that the work function of the W-matrix is lowered by the presence of UC but not to the value of the UC work function. The study is being continued at temperatures higher than 1550°C.

### 3. Life Testing Fueled Cesium Thermionic Converters

The main emphasis was in the continued development effort of a cylindrical fuel emitter structure and in the preparations of the testing equipment. In the meantime the collector and envelope parts for the first converter have been fabricated and await assembly to the emitter structure. Under the assumption of a successful emitter development, the first converter test is scheduled to begin in the second week of March. Details of the January progress are discussed in the following paragraphs.

#### 3.1 Fueled Emitter Fabrication Development

Two fuelled emitter design concepts were described in the December progress report which would eliminate diffusion bonding the tantalum stem onto the fueled emitter body. This would avoid emitter cracking resulting from stress concentrations at fuel slots and thermocouple holes. Such cracking was observed in the first emitter made for this program.

One of the designs (see Fig. 3-1, December, 1963 monthly report<sup>(2)</sup>) was mocked up with an unfueled emitter slug coated with 0.040 mil of tungsten. A satisfactory, leak tight, diffusion-bonded joint was made. This was machined to final dimensions and thermally cycled with no degradation of the joint. Following this successful test an actual blank was machined and fabrication of the emitter is awaiting the preparation of fuel slabs (see below).

Fabrication of an emitter of the design illustrated in Fig. 3-2 of December, 1963 monthly report<sup>(2)</sup> was also commenced. The



tantalum was satisfactorily bonded to the tungsten and machining was carried to the point of making the slots for the fuel. This is being held upon pending receipt of the fuel slabs, so the slots can be custom-made for a tight, slip fit.

Cylinders of stoichiometric 30 mol-% UC-70 mol-% ZrC were isostatically pressed and sintered to provide material for fabrication of the fuel slabs. Fourteen slabs (six are required for each emitter) of the required design were successfully machined without breakage. This was a large improvement over the difficulties encountered in making the fuel slabs for the first emitter.

To prevent attack of the carbides while the final 0.042 in. tungsten clad is being applied to the emitter, a protective seal coat, 0.003 in. thick, was applied. The coatings looked excellent, but when one slab was sectioned and examined metallographically, attack of the carbide by the coating process was noted. Several other specimens were then sectioned, and all showed attack. Experimentation related to elimination of this attack is described in Section 1.2 above. While progress has been made, satisfactory seal coats are not yet available. Additional carbide slabs have been prepared to insure their readiness when satisfactory coatings can be applied. Further work related to emitter development must wait until coated fuel slabs are available.

### 3.2 Design

Converters for the life testing of fuel bearing electrically heated emitters are design-wise designated the Mark VI-E. The preliminary design was shown in Fig. 3-4 of the first quarterly report of this contract<sup>(3)</sup>.

Design details have been completed and the converter assembly and parts drawings approved.

The design layout of the test stand, including supports, current leads, heating and cooling lines has been completed. The detail drawings are being completed and checked. Machining of these parts will be started during the week of February 10.

The collector heat sink design shown in Fig. 3-1, provides for both heating and cooling of the collector. The heat sink is made of molybdenum to match the thermal expansion of the collector that is taper fitted to the I.D. of the heat sink.

In the first assembly attempt an Inconel-sheathed nichrome heater was copper brazed to the heat sink. The stainless steel cooling coil was subsequently brazed with nickel-gold. Because of the thermal expansion mismatch between Inconel and molybdenum, the heater sheath has been changed to tantalum and the braze made with a paladium-cobalt alloy (Weago Palco). The stainless steel cooling coil will be brazed with copper-titanium.

### 3.3 Test Apparatus

All the equipment necessary for the test stands for the life test has been ordered, except for the emitter temperature controllers which are short lead time items. One of these is undergoing performance evaluation at this time. All equipment for one stand is expected to be received during March. A three week completion time for assembly and check out of the equipment is anticipated. The first test stand will be in operation before April 1, provided the vendors meet their delivery schedule. The automatic data logging system has a delivery date of April 1.

Two weeks for hook-up and check out time are also anticipated for this equipment.

The first converter will be operated in the General Atomic owned Varian Vac-Ion vacuum stand if converter fabrication is completed prior to March 1. Converters completed after March 13 will be prepared for installation into the new test stands.

#### 4. Irradiation Studies

A trip was made to P. B. R. F. in early January to discuss the document accompanying the request for irradiation submitted in December and to examine the reactor arrangement during shutdown. The visit was very successful as our assigned project engineer, Mr. Gary Snyder, was very helpful in constructively criticizing our document so that it could be most easily altered to become the final safeguards and design manual. At that time the guide for preparation of this final document was examined and discussed in detail.

A tour of reactor arrangement was made so that accessibility of coolant water lines, accessory equipment space, and routing of instrumentation and control leads could be determined for future design work. It appears that use of some of the space in the air annulus beneath the missile shield to locate remotely-operable coolant valves, flow controls and alarms, and other accessory equipment is feasible and advantageous. A section of the steel grating immediately adjacent to the assigned V-tube location may need slight modification for access. Gas cylinders (helium and nitrogen for temperature control) will probably be located on the mezzanine below the first floor. Instrumentation leads can be routed through any of several adjacent trays to a suitable penetration and thence to the experiment control room.

The Mock-Up Reactor was visited to physically observe the arrangement in anticipation of performing the proposed gold foil activation experiments. This group of experiments were discussed with Snyder to further explain their purpose and value. The problem of foil holding and the accuracy of their location was examined in council with the P. B. R. F. physicists.

It was tentatively decided that the foil holders for the in-core foils was usable but a new holder might be required to suit the specific experiments and locations within the V-tube.

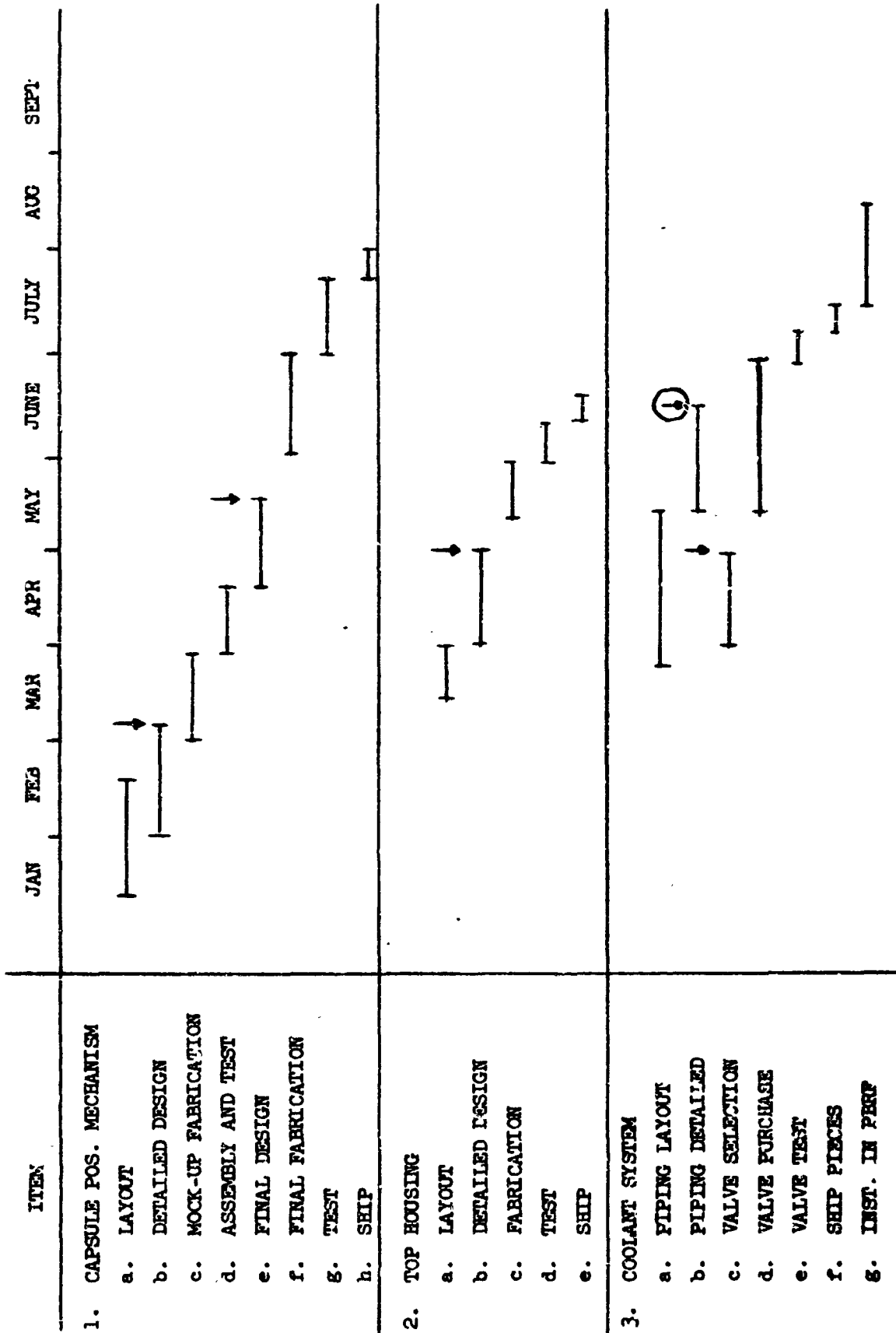
The design work has progressed to the layout of the capsule positioning mechanism and the hydraulic cable drive system. In order to proceed with the design of the V-tube and top housing, the following specifications of design parameters had to be established.

- (a) The V-tube will be operated at primary system pressure and full of water.
- (b) Pressure drop calculations indicate that sufficient head for the experiment is available between the reactor primary inlet and return. A coolant pump will no longer be required.
- (c) Connections to the primary water inlet and return headers can be made in the air annulus beneath the missile shield. Control valving, remotely operated, may also be housed in this space.
- (d) An opening in the V-tube will be provided for transfer of an irradiated capsule to a cut-off tool temporarily mounted on the reactor vessel wall. A decision by P. B. R. F. personnel has been promised on the necessity of a door for this opening.
- (e) The capsule positioning mechanism will be driven by 1/8 in. stainless steel cables actuated by a hydraulic piston. Since the actuating mechanism will be located in a housing flanged to the V-tube and bathed in water, it will be made from stainless steel (probably type 316).

- (f) The motion and direction of the capsule positioning mechanism (CPM) will be regulated by a hydraulic servo system controlled by electrical relays. The relays are actuated on signals from the temperature recording instrument. The position of the capsule will be indicated by mechanical switches operating relays to transmit electrical signals to an event recorder.
- (g) The final design layout of the CPM has been completed and details are underway. The structural calculations indicate that the design can be modified to simplify assembly and disassembly. A plastic model of the CPM will be built to evaluate the design.

The effort on the various Plum Brook hardware requirements has been scheduled as shown in the attached bar chart.

PRELIMINARY SCHEDULE OF PLAM BROCK CAPSULE EQUIPMENT MODIFICATIONS



ITEM	JAN	FEB	MAR	APR	MAY	JUNE	JULY	AUG	SEPT
4. V-TUBE MODIFICATION									
a. PRELIMINARY DESIGN									
b. FINAL DESIGN									
c. PURCHASE									
d. TEST WITH C.P.M.									
e. SHIP									
	COMPLETE								
	V-TUBE FINAL DESIGN; PURCHASE AND TEST NOT YET AUTHORIZED								
5. CAPSULE DESIGN									
a. PRELIMINARY DESIGN									
b. FINAL DESIGN									
c. FUEL FABRICATION									
d. ASSEMBLY									
e. SHIP									
	COMPLETE								
	AWAITS MUR EXP. RESULTS - MUR EXP. NOT YET AUTHORIZED								
6. MANUAL PREPARATION									
HAZARDS									
DESIGN									
OPERATING									

↓ INDICATES TARGET DATE FOR SUBMISSION OF DRAWINGS TO PROJECT ENGINEER

⬇ INDICATES REQUIREMENT FOR DECISION ON WHO DOES INSTALLATION AT PERP

--- INTERMITTENT EFFORT AS REQ'D



REFERENCES

- (1) Final Report of Contract NAS 3-2532, GA-4769.
- (2) December Monthly Report of Contract NAS 3-4165, GACD-4923.
- (3) First Quarterly Report of Contract NAS 3-4165, GA-4874.

TABLE 1-1

BET Measurements of UC-ZrC Specimens

Composition	Powder Fraction Employed	Sintering Temp. (°C)	Density (% Theoretical)	Roughness Factor
90 UC - 10 ZrC	-45 $\mu$	1900	90.3	62
90 UC - 10 ZrC <sup>(a)</sup>	-45 $\mu$	1900	90.3	75
90 UC - 10 ZrC	-45 $\mu$ /+20 $\mu$	1900	83.1	300
90 UC - 10 ZrC	-45 $\mu$ /+20 $\mu$	1900	83.0	360
UC	-45 $\mu$ /+20 $\mu$	2000	85.0	188
UC	-45 $\mu$ /+10 $\mu$	2000	87.0	98

NOTE: All specimens isostatically pressed at ~ 97,000 psi

(a) Same specimen as specimen listed above in table, but with a 1/4" diameter hole cut axially through specimen.

Spectrographic Analysis of  $WF_6$ 

<u>Element</u>	<u>Concentration (ppm)</u>	<u>Element</u>	<u>Concentration (ppm)</u>
Al	6	Na	N<20
As	N<20 <sup>(a)</sup>	Ni	N< 1
B	N< 4	Pb	N< 4
Ba	N< 2	Sb	N< 6
Be	N< 1	Si	< 2
Bi	N< 1	Sn	N< 2
Cd	N< 8	Sr	N<20
Co	N< 1	Te	N<200
Cr	N<10	Tl	8
Cu	<0.5	Tl	N< 8
Fe	8	V	N< 8
Hg	N< 8	Zn	N<20
In	N<20	Zr	N<200
Mg	1		
Mn	N<0.5		
Mo	N<200		

(a) N means not detected and the number is the limit of detection.

TABLE 2-1

Vapor Loss Investigation of Cold-Pressed and Sintered  
29.4 Mol% UC-70.6 Mol% ZrC Sample A<sub>2</sub>-1 (77.6% dense)

Run Number	Temperature (°K)	Exposure Time (sec x 10 <sup>4</sup> )	Rate of Weight Loss (mg/cm <sup>2</sup> /sec) x (10 <sup>-5</sup> )	Density gms/cm <sup>3</sup>
Original	-	-	-	6.99
Degas	2248	4.80	21.2	7.01
1	2284	7.20	2.61	7.00
2	2191	7.65	1.48	7.00
3	2093	10.08	0.266	7.01

TABLE 2-2

Fission Product Release at 1600°C from sample A<sub>2</sub>-3 (77.8% dense, 29.4. UC-70.6 ZrC)

Temp. °C	Time (hr.)	Fractional Release			
		Xe <sup>133</sup>	Te <sup>132</sup>	I <sup>131</sup>	Ba <sup>140</sup>
1600	0 (reccl)	1.9 x 10 <sup>-3</sup>	-	-	-
	18.24	0.014	0.18	0.04	0.0077
	41.80	0.10	0.50	0.17	0.0145
	67.00	0.16	0.62	0.25	0.027

TABLE 2-3

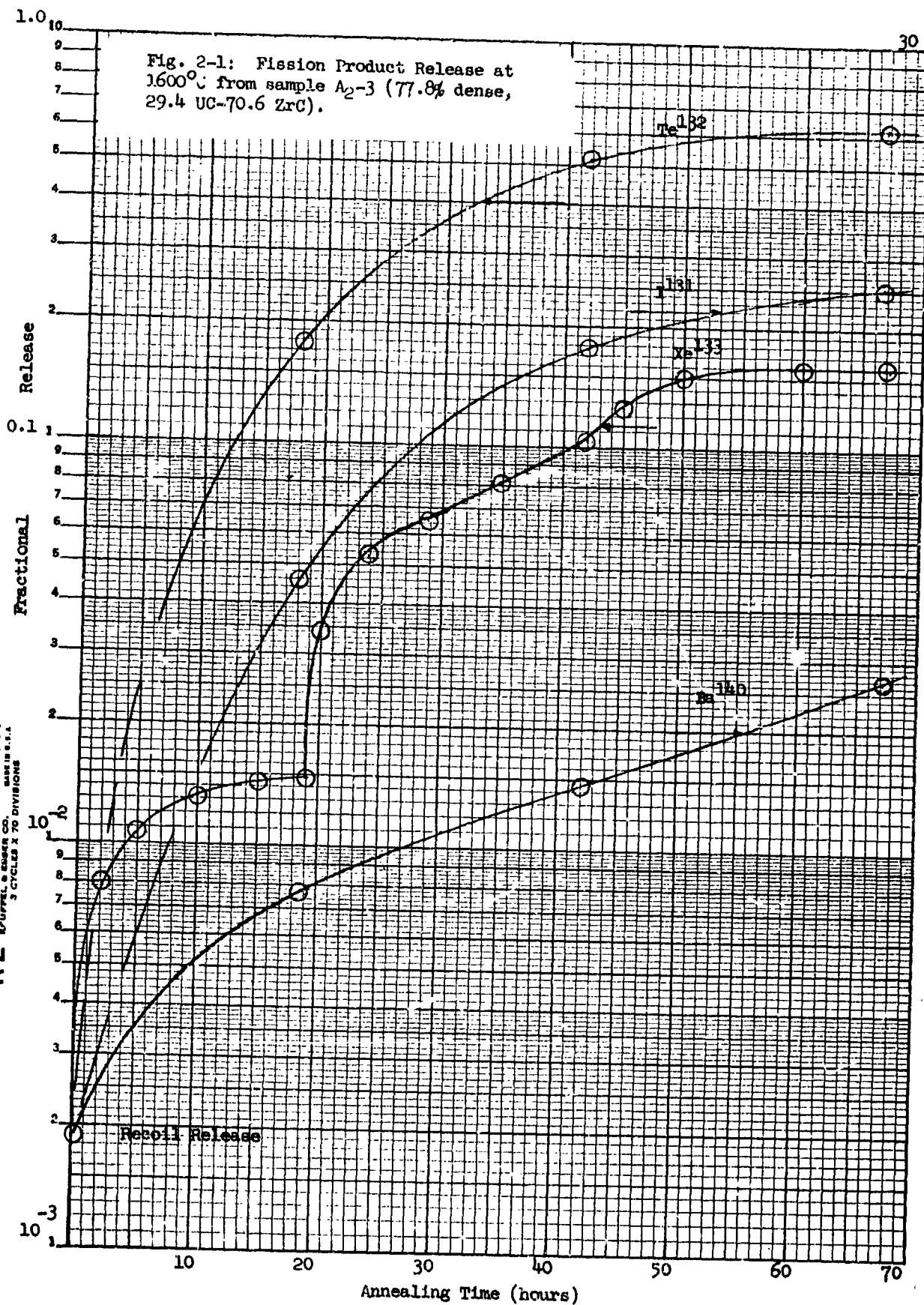
Gross Diffusion Experiments on Fuel-Clad Systems

Experiment	Temperature	Time	Fuel Composition	Clad Materials	Present Status
D <sub>1</sub>	1800°C	50 hours	30 UC - 70 ZrC (U 0.134 <sup>C</sup> Zr 0.367 <sup>C</sup> 0.499±0.005)	Mo Ir Re W-25Re	Reported in December, 1963 Report (2).
D <sub>2</sub>	1600°C	50 hours	30 UC - 70 ZrC (U 0.134 <sup>C</sup> Zr 0.367 <sup>C</sup> 0.499±0.005)	Mo Ta Nb	Preliminary Metallo- graphical examinations made. See Text.
D <sub>3</sub>	1600°C	50 hours	30 UC - 70 ZrC (U 0.134 <sup>C</sup> Zr 0.367 <sup>C</sup> 0.499±0.005)	Mo Ir Re W-25Re	Same as above
D <sub>4</sub>	1600°C	50 hours	UC (U 0.499 <sup>C</sup> 0.501±0.005)	Mo Nb Ta Re	Same as above
D <sub>5</sub>	1600°C	100 hours	30 UC - 70 ZrC (U 0.150 <sup>C</sup> Zr 0.346 <sup>C</sup> 0.504±0.004)	Mo Re	Experiment completed Samples to be examined.
D <sub>6</sub>	1400°C	100 hours	Same as above	Mo Ta Nb	Experiment completed Samples to be examined.

TABLE 2-4

Refractory Metals Gross Diffusion Experiments

Experiment	Temperature	Time	Diffusion Couple	Present Status
F <sub>1</sub>	1800°C	50 hours	Tungsten vs molybdenum	Preliminary results reported in Dec. 1963 report <sup>(2)</sup>
F <sub>2</sub>	1600°C	50 hours	Tungsten vs niobium Tungsten vs tantalum	Being studied by microprobe
F <sub>3</sub>	1600°C	50 hours	Tungsten vs tantalum  Tungsten vs niobium Tungsten vs molybdenum	See text, section 2.6





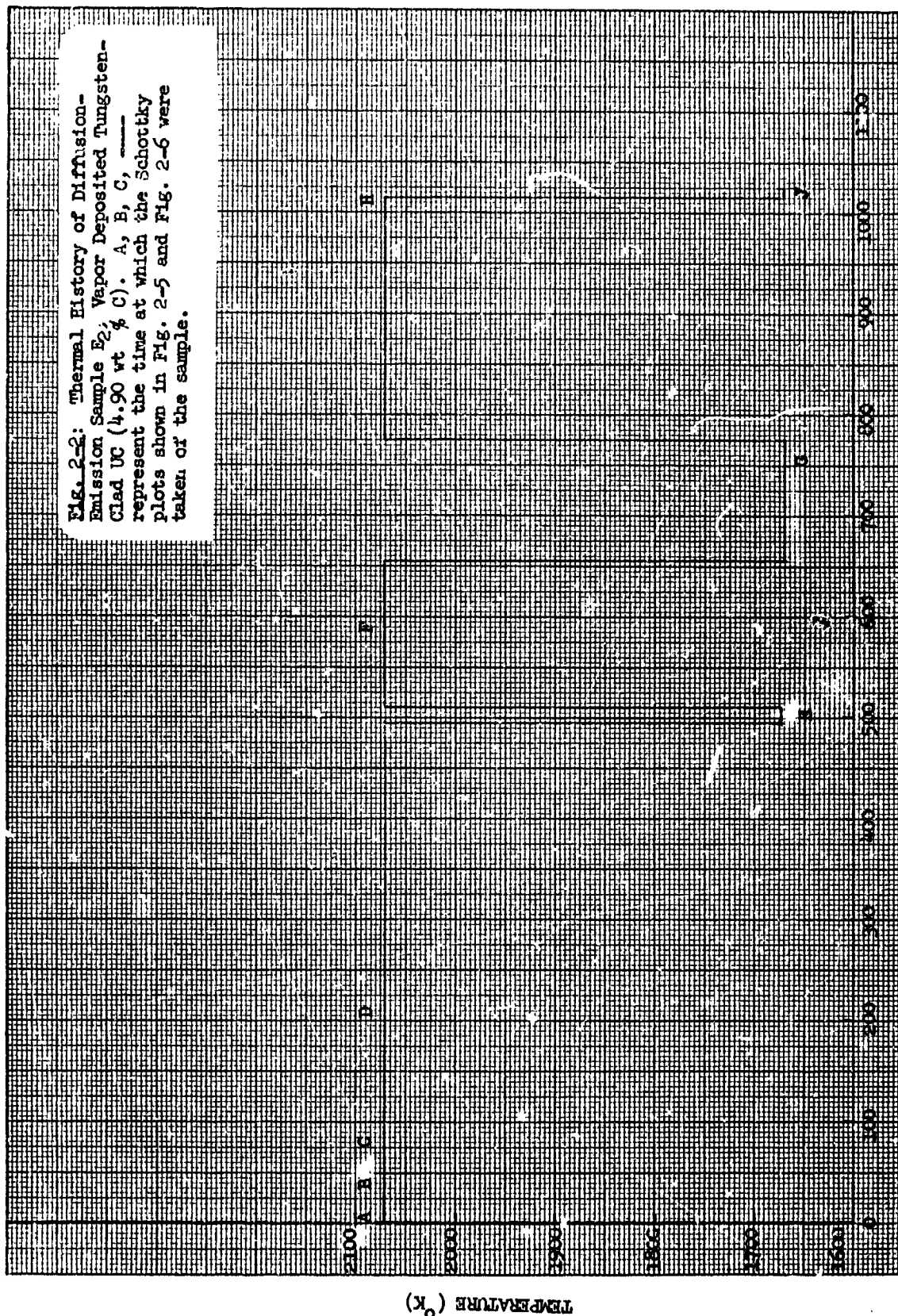
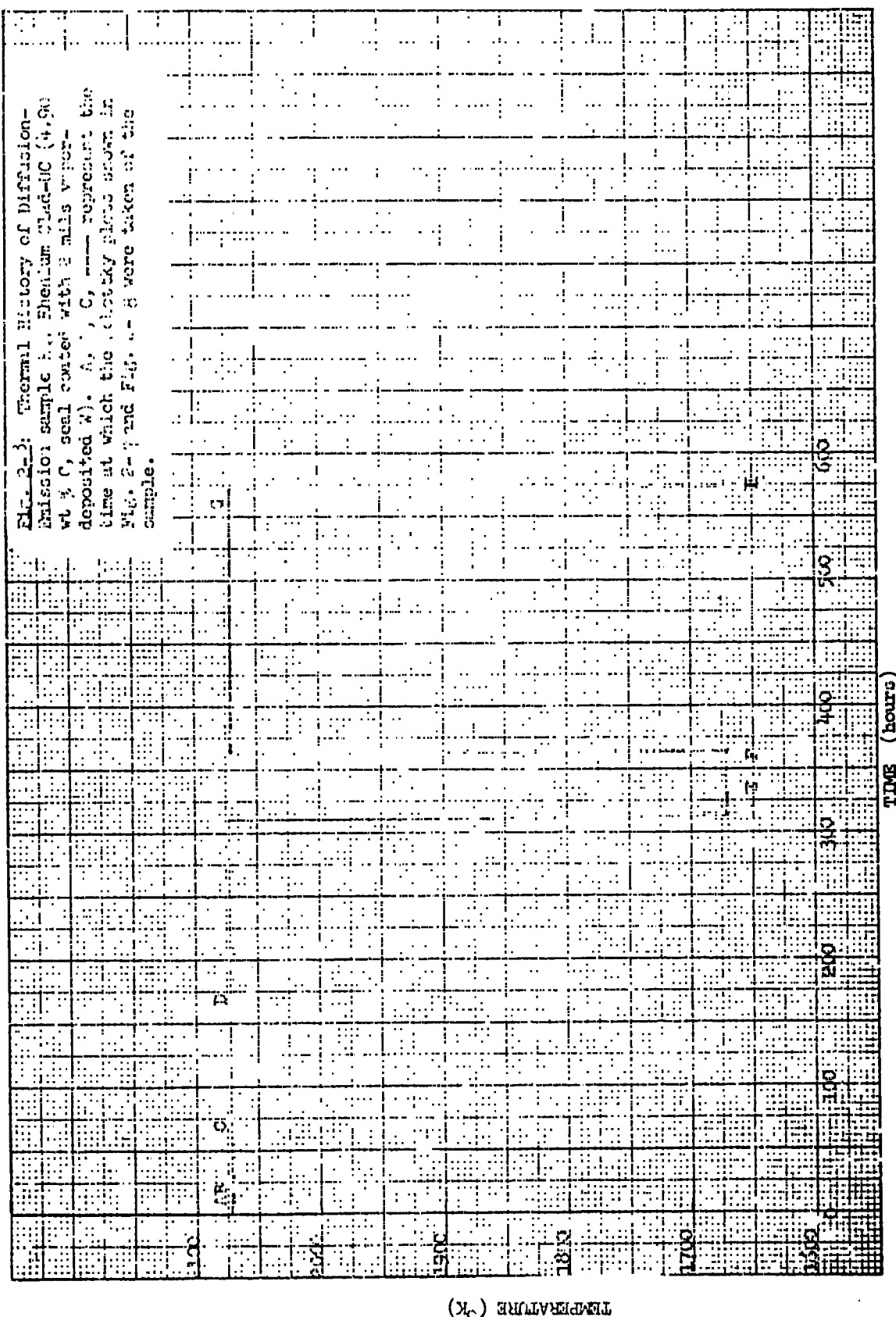


Fig. 2-2: Thermal History of Diffusion-Emission Sample E2; Vapor Deposited Tungsten-Clad UC (4.90 wt % C). A, B, C, --- represent the time at which the Schottky plots shown in Fig. 2-5 and Fig. 2-6 were taken of the sample.



K-E 10 X 10 TO THE 1/4 INCH 3SP-11  
KEUPPEL & EBER CO. HANNOVER, N.H.

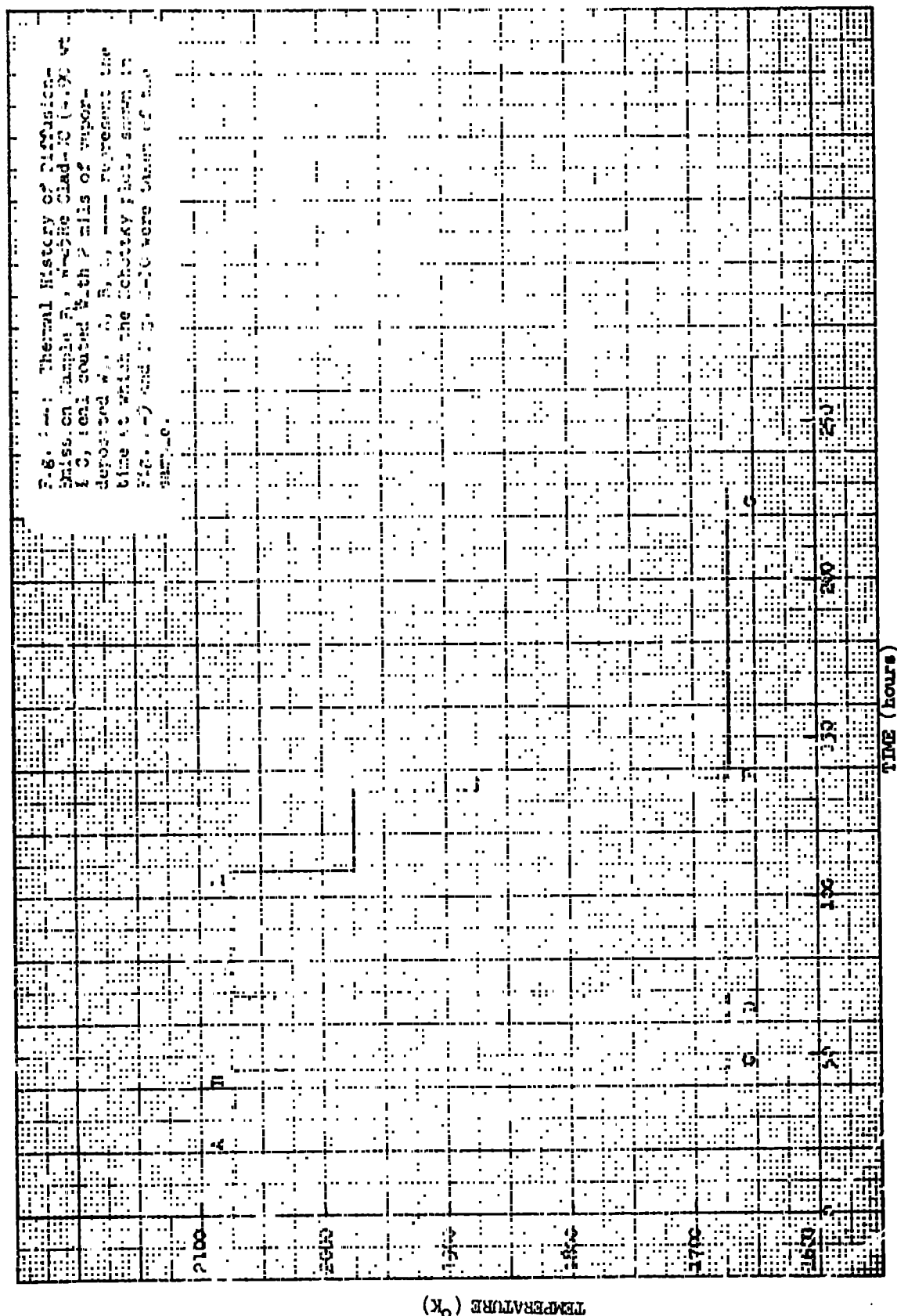
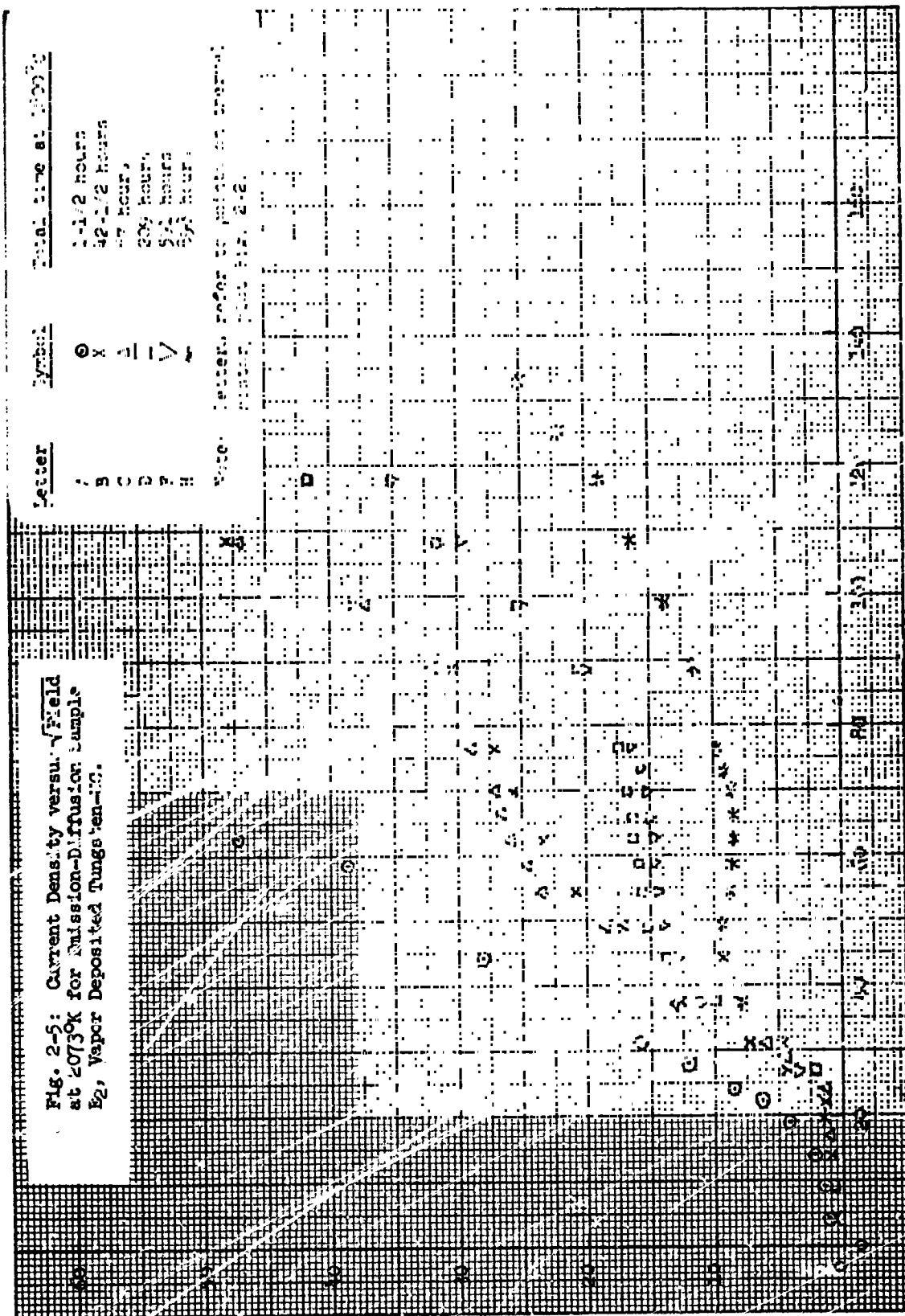


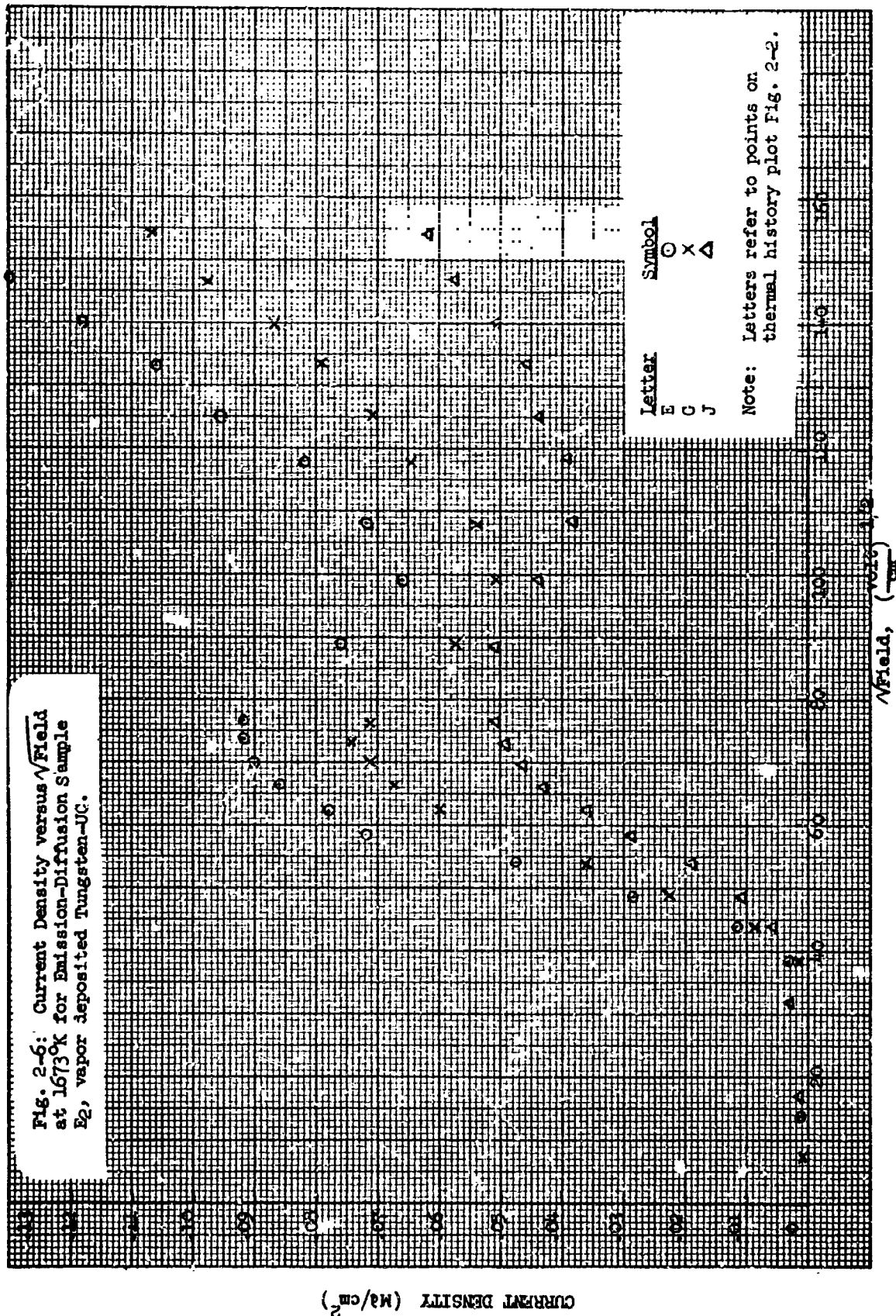
Fig. 2-5: Current Density versus  $\sqrt{\text{Field}}$   
at  $\pm 073^\circ\text{K}$  for Emission-Diffusion Sample  
 $\text{E}_2$ , Vapor Deposited Tungsten-10.



CURRENT DENSITY (mA/cm²)

$\sqrt{\text{Field}}, \left(\frac{\text{VOLT}}{\text{CM}}\right)^{1/2}$

Fig. 2-6: Current Density versus  $\sqrt{\text{Field}}$   
at 1673°K for Emission-Diffusion Sample  
E<sub>2</sub>, vapor deposited Tungsten-UC.



Letter	Symbol	Total time at 1800°C
A	○	2 1/2 hours
B	x	20 1/2 hours
C	△	69 hours
D	□	173 hours
G	*	515 hours

Note: Letters refer to points on thermal history plot Fig. 2-3.

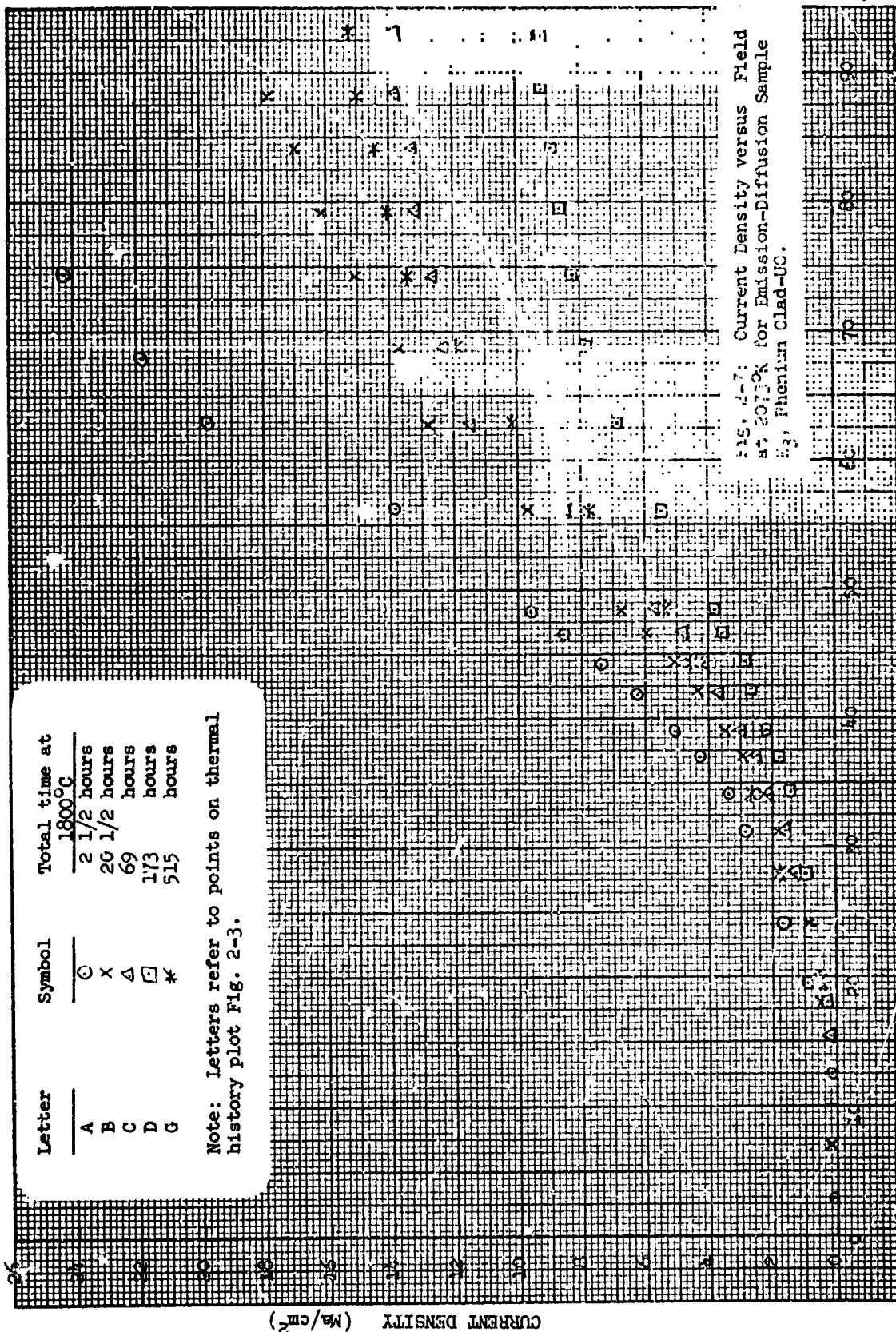


Fig. 2-7: Current Density versus Field at 201°C for Emission-Diffusion Sample #3, Phenium Clad-UC.

Field (volt/cm)<sup>1/2</sup>



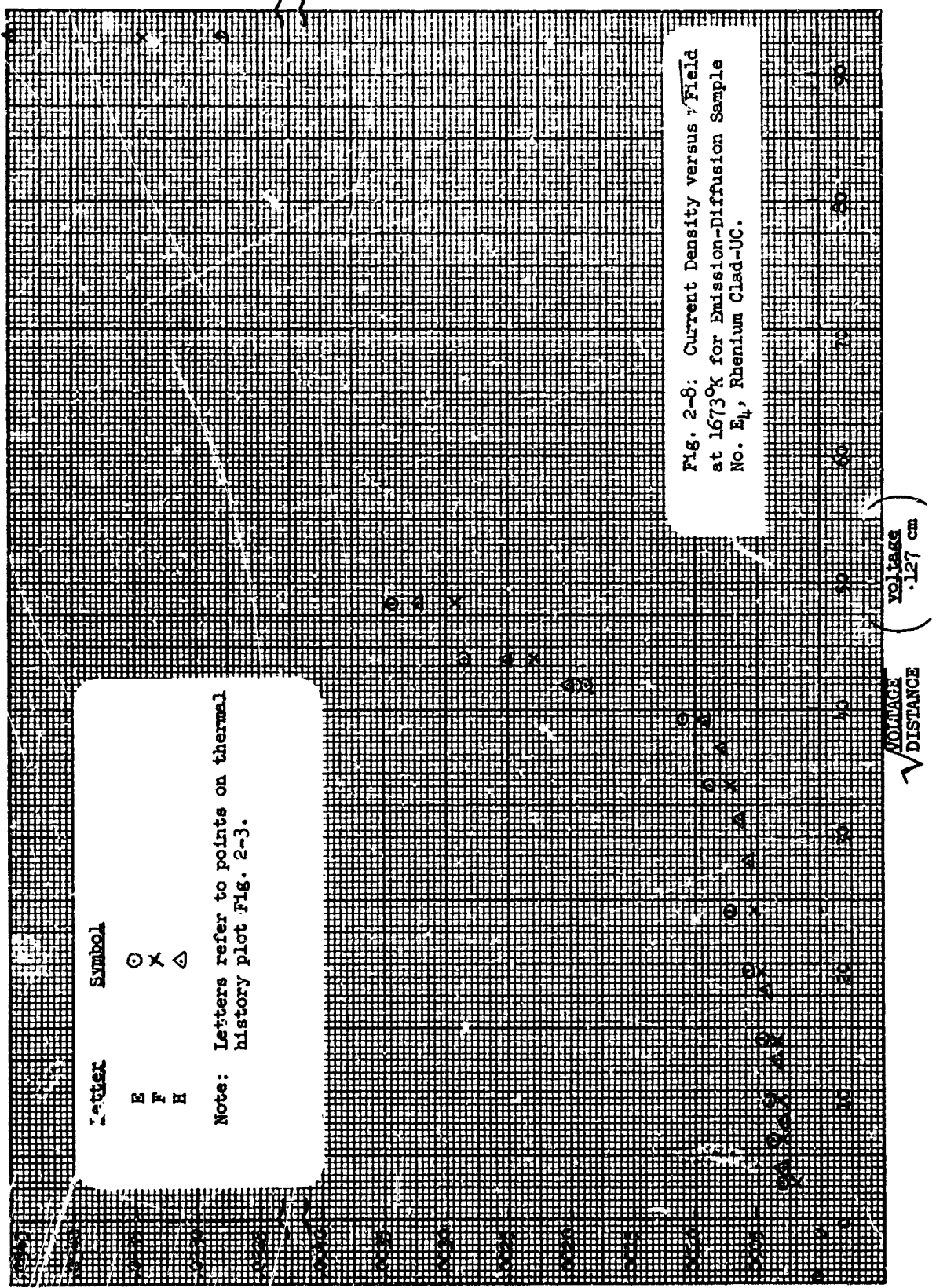
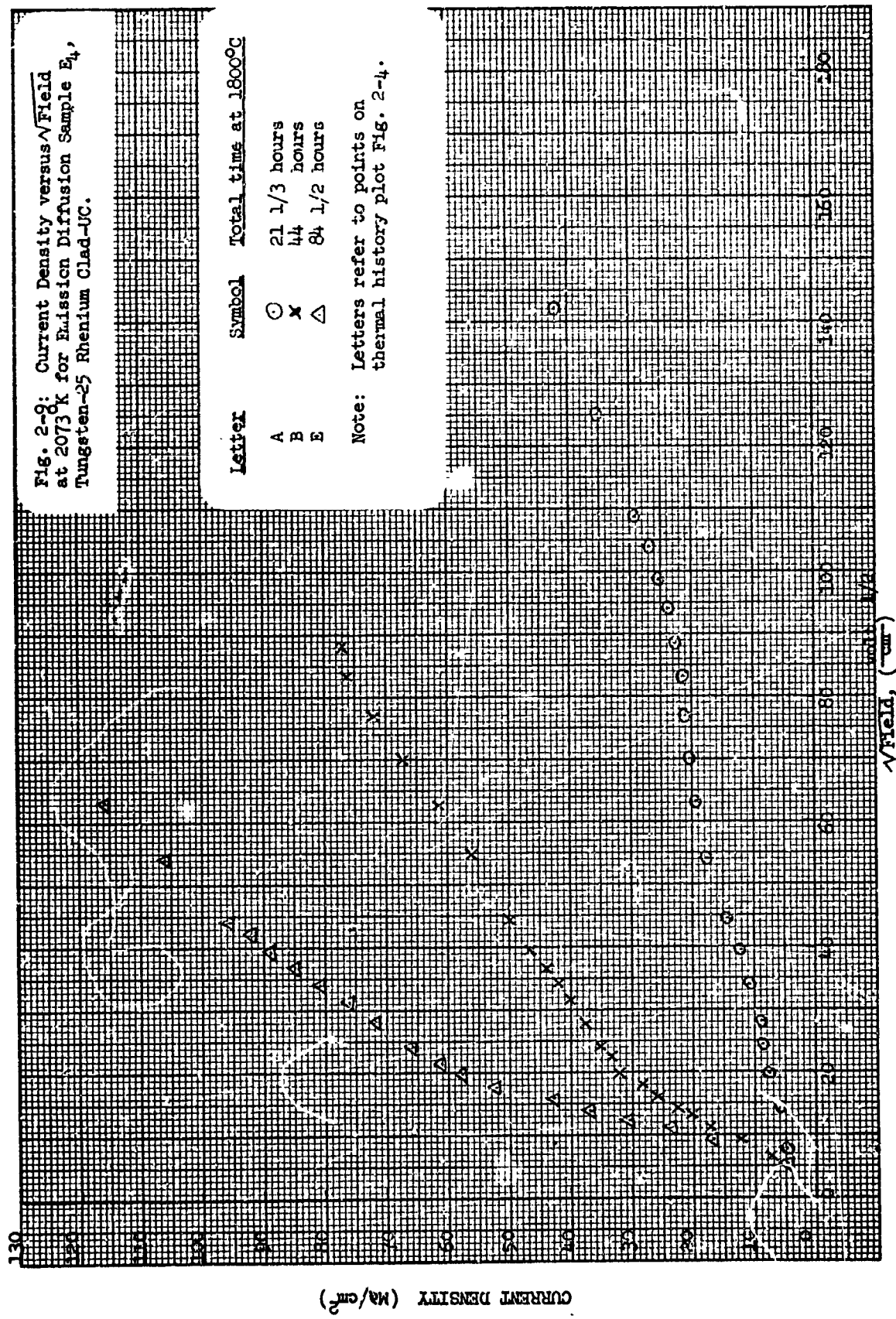
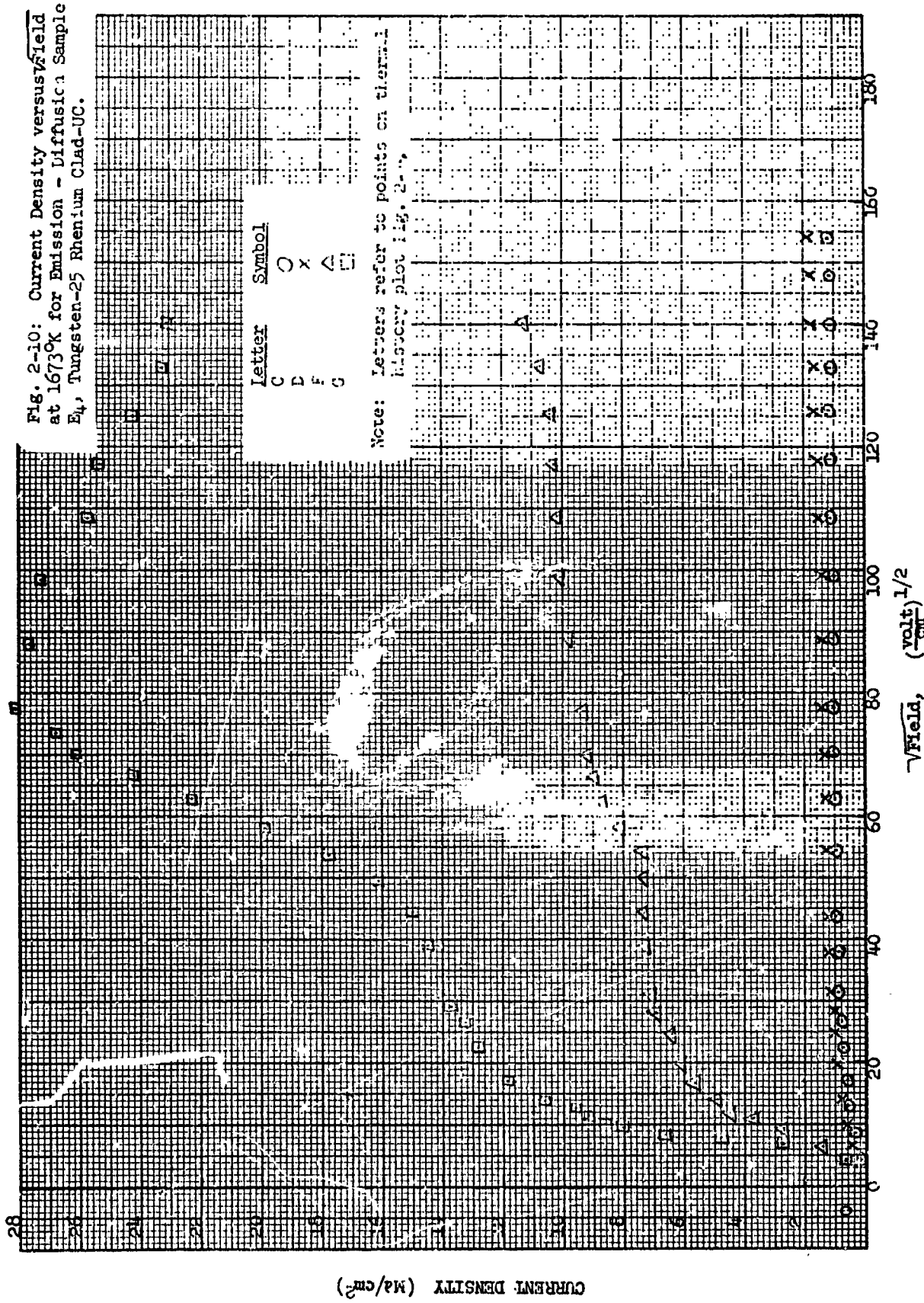


Fig. 2-3: Current Density versus  $\sqrt{\text{Field}}$   
 at 2073 K for Emission Diffusion Sample  $E_1$ ,  
 Tungsten-25 Rhenium Clad-UC.







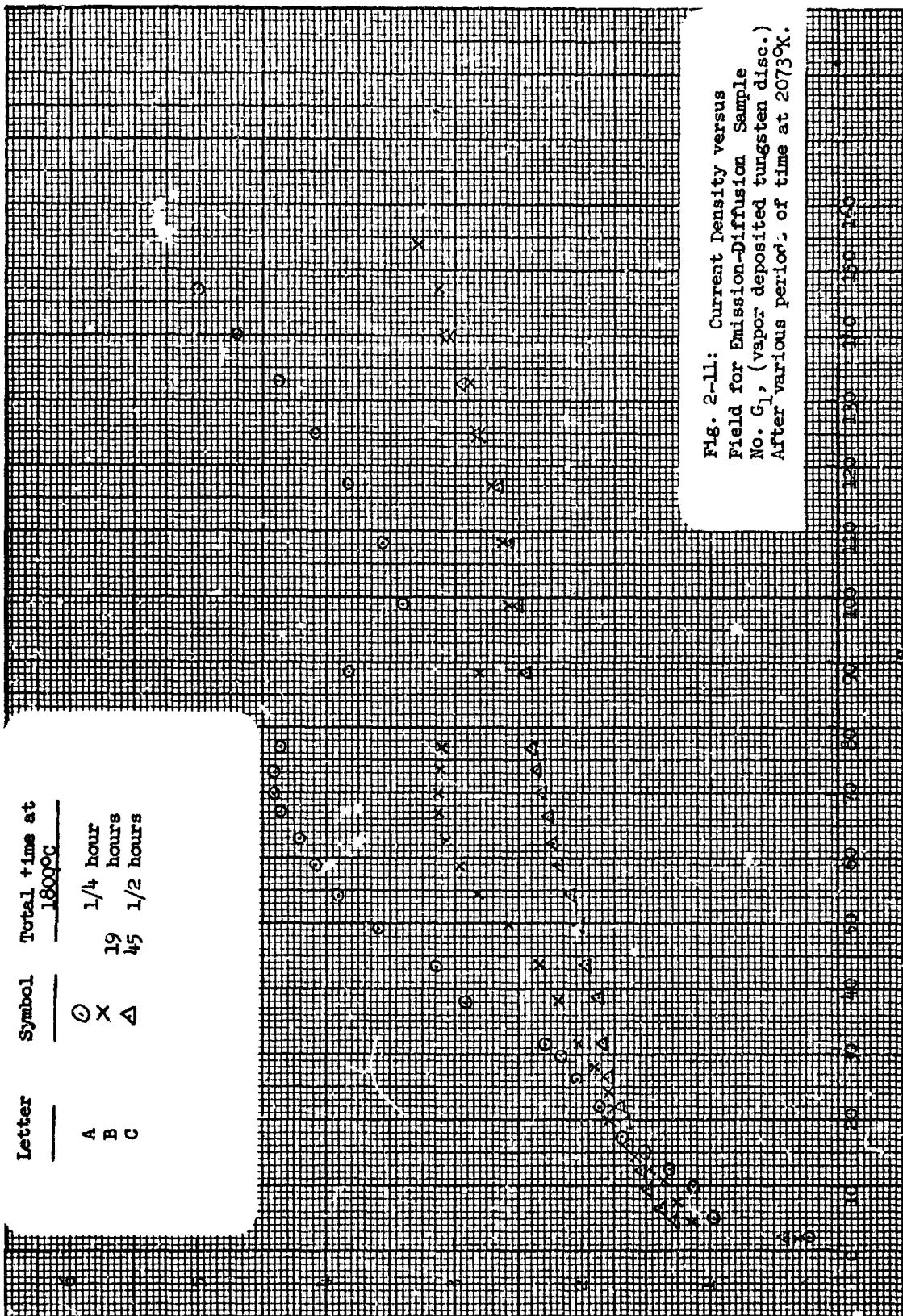
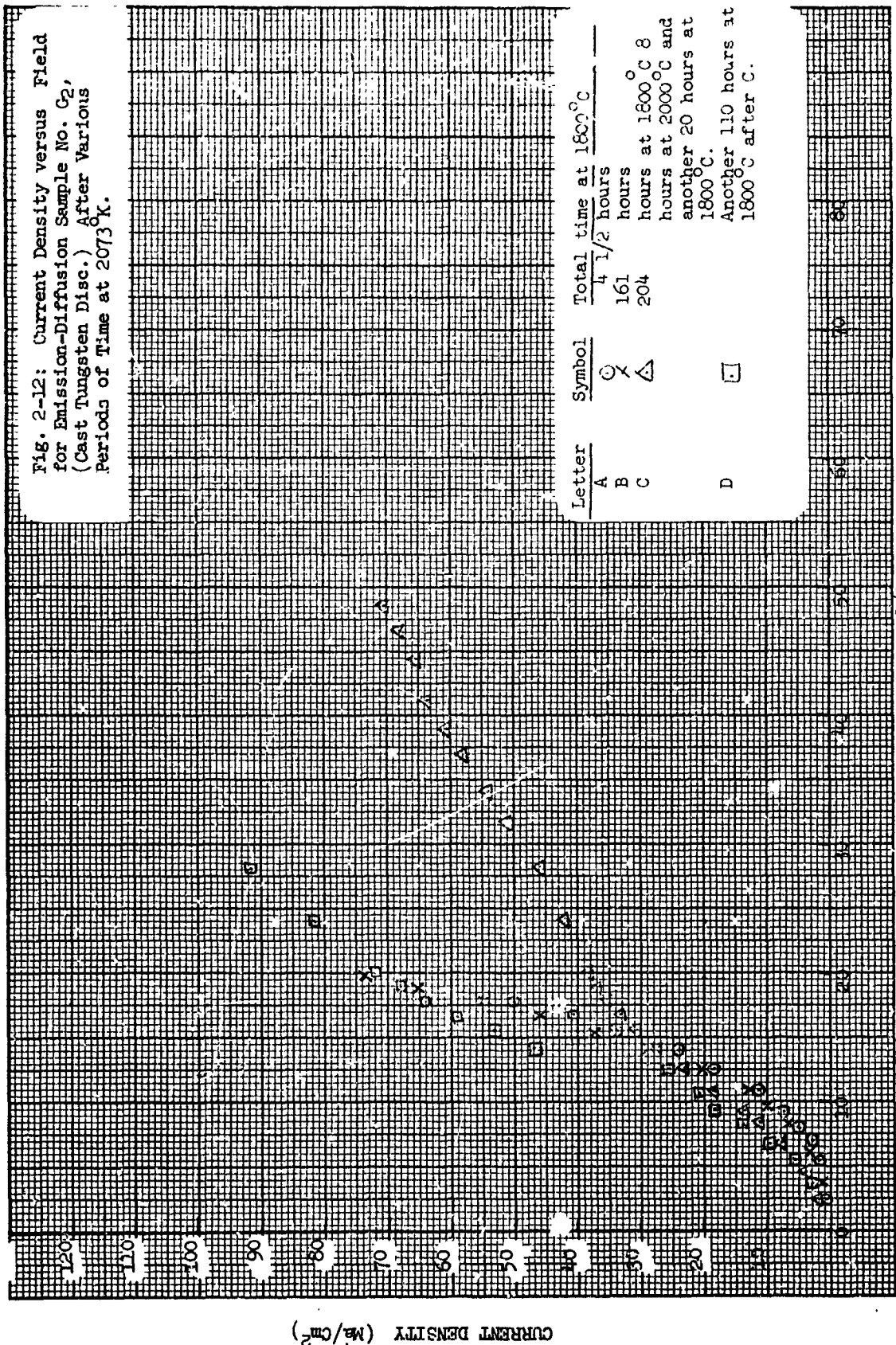
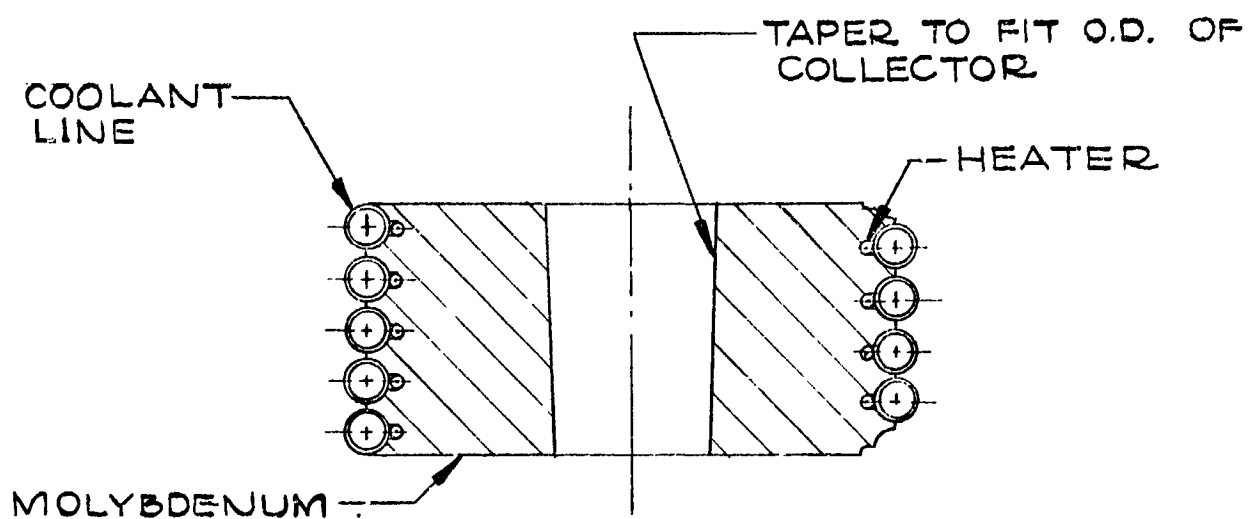


Fig. 2-11: Current Density versus  
 Field for Emission-Diffusion Sample  
 No. G<sub>1</sub>, (vapor deposited tungsten disc.)  
 After various periods of time at 2073°K.





## COLLECTOR HEAT SINK

Fig. 3-1